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Copper and copper alloys - Determination of chromium content - Part 2: FAAS method

Kupfer und Kupferlegierungen - Bestimmung des Chromgehaltes - Teil 2:
Flammenatomabsorptionsspektrometrisches Verfahren (FAAS)

Cuivre et alliages de cuivre - Dosage du chrome - Partie 2. Méthode par spectrométrie
d'absorption atomique dans la flamme (SAAF)

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Ta slovenski standard je istoveten z: EN 14940-2:2006

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English Version

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Cuivre et alliages de cuivre - Dosage du chrome - Partie 2 :
Méthode par spectrométrie d'absorption atomique dans la
flamme (SAAF)

Kupfer und Kupferlegierungen - Bestimmung des
Cobaltgehaltes -
Flammenatomabsorptionsspektrometrisches Verfahren
(FAAS)

This European Standard was approved by CEN on 15 May 2006.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 14940-2:2006) has been prepared by Technical Committee CEN/TC 133 "Copper and copper alloys", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2006, and conflicting national standards shall be withdrawn at the latest by December 2006.

Within its programme of work, Technical Committee CEN/TC 133 requested CEN/TC 133/WG 10 "Methods of analysis" to prepare the following standard:

EN 14940-2, *Copper and copper alloys — Determination of chromium content — Part 2: FAAS method*

This is one of two parts of the standard for the determination of chromium content in copper and copper alloys. The other part is:

prEN 14940-1, *Copper and copper alloys — Determination of chromium content — Part 1: Titrimetric method*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This part of this European Standard specifies a flame atomic absorption spectrometric method (FAAS) for the determination of the chromium content of copper and copper alloys in the form of unwrought, wrought and cast products.

The method is applicable to products having chromium mass fractions between 0,010 % and 2,0 %.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1811-1, *Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 1: Sampling of cast unwrought products*

ISO 1811-2, *Copper and copper alloys — Selection and preparation of samples for chemical analysis — Part 2: Sampling of wrought products and castings*

NOTE Informative references to documents used in the preparation of this standard, and cited at the appropriate places in the text, are listed in the Bibliography.

3 Principle

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Dissolution of a test portion in nitric acid and digestion in sulphuric acid followed, after suitable dilution, by aspiration into an air-acetylene or into a nitrous oxide-acetylene flame of an atomic absorption spectrometer. Measurement of the absorption of the 357,9 nm line emitted by a chromium hollow-cathode lamp.

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4 Reagents and materials

4.1 General

During the analysis use only reagents of recognised analytical grade and only distilled water or water of equivalent purity.

4.2 Sulphuric acid, H₂SO₄ ($\rho = 1,84$ g/ml).

4.3 Hydrogen peroxide, H₂O₂ 30 % (mass fraction) solution.

4.4 Nitric acid, HNO₃ ($\rho = 1,40$ g/ml).

4.5 Nitric acid solution, 1 + 1

Dilute 500 ml of nitric acid (4.4) in 500 ml of water.

4.6 Hydrofluoric acid, HF ($\rho = 1,13$ g/ml).

WARNING — Hydrofluoric acid is a hazardous substance. Care shall be taken and it shall be used under an efficient fume hood.

4.7 Chromium stock solution, 0,500 g/l Cr

Weigh 1,414 2 g of potassium dichromate ($K_2Cr_2O_7 > 99,9 \%$), previously dried at $140 \text{ }^\circ\text{C}$ and allowed to cool in a desiccator. Place in a 400 ml beaker and dissolve in about 20 ml of water. Add 5 ml of sulphuric acid (4.2) and, while cooling, cautiously add the hydrogen peroxide solution (4.3), adding an excess of about 2 ml after effervescence has ceased. Allow the solution to stand at ambient temperature until the yellow colour has completely disappeared (several hours), then transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contains 0,500 mg of Cr.

4.8 Chromium standard solution, 0,025 g/l Cr

Transfer 25,0 ml of the chromium stock solution (4.7) into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

Prepare this solution immediately prior to use.

1 ml of this solution contain 0,025 mg of Cr.

4.9 Copper base solution, 20 g/l Cu

Weigh 20,0 g of chromium-free copper ($Cu \geq 99,90 \%$, $Cr \leq 0,001 \%$) and transfer into a 1 000 ml beaker. Add, in small portions, 400 ml of nitric acid solution (4.5). Cover the beaker. If necessary, heat gently to help dissolution. After cooling, add cautiously 200 ml of the sulphuric acid (4.2); heat to the evolution of copious white fumes and continue for 5 min. Cool and add cautiously about 200 ml of water and heat to complete the dissolution. Cool to room temperature, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

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5 Apparatus

5.1 Ordinary laboratory apparatus.

5.2 Atomic absorption spectrometer, fitted with an air-acetylene or a nitrous-oxide acetylene burner.

5.3 Chromium hollow-cathode lamp.

6 Sampling

Sampling shall be carried out in accordance with ISO 1811-1 or ISO 1811-2, as appropriate.

Test samples shall be in the form of fine drillings, chips or millings with a maximum thickness of 0,5 mm.

7 Procedure

7.1 Preparation of the test portion solution

7.1.1 Test portion

Weigh ($1 \pm 0,001$) g of the test sample.

7.1.2 Chromium mass fractions between 0,01 % and 0,20 %

Transfer the test portion (7.1.1) into a 250 ml beaker. Add 20 ml of the nitric acid solution (4.5) and 0,5 ml of hydrofluoric acid (4.6), cover and heat gently until the test portion is completely dissolved. Allow to cool and cautiously add 20 ml of the sulphuric acid (4.2). Heat on a sand bath at a temperature between 230 °C and 240 °C for at least 4 h. Take care in the evaporation not to go under 20 ml. Allow to cool, add 20 ml to 30 ml of water and heat to dissolve the salts.

Transfer the test solution into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

7.1.3 Chromium mass fractions between 0,20 % and 2,0 %

Transfer the test portion (7.1.1) into a 250 ml beaker. Add 20 ml of the nitric acid solution (4.5) and 0,5 ml of hydrofluoric acid (4.6), cover and heat gently until the test portion is completely dissolved. Allow to cool and cautiously add 20 ml of the sulphuric acid (4.2). Heat on a sand bath at a temperature between 230 °C and 240 °C for at least 4 h. Take care in the evaporation not to go under 20 ml. Allow to cool, add 20 ml to 30 ml of water and heat to dissolve the salts.

Transfer the test solution into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix well. Transfer 10,0 ml of this solution into another 100 ml one-mark volumetric flask. Add 45 ml of the copper base solution (4.9). Dilute to the mark with water and mix well.

7.2 Blank test

Carry out a blank test simultaneously with the determination, following the same procedure and using the same quantities of all reagents as used for the determination, but omitting the test portion.

7.3 Check test

Make a preliminary check of the apparatus by preparing a solution of a standard material or a synthetic sample containing a known amount of chromium and of composition similar to the material to be analysed. Carry out the procedure specified in 7.5.

7.4 Establishment of the calibration curve

7.4.1 Preparation of the calibration solutions

7.4.1.1 General

In all cases, copper, chloride and nitrate concentrations and acidity in the calibration solutions shall be similar to those of the test portion solutions.

The presence of copper in the standard calibration solutions compensates for chemical interaction effects of copper in the test portion solution. Normally no similar additions are required to compensate for the effects of alloying elements. If any alloying element is present in the material to be analysed in mass fraction > 10 % an appropriate mass of this element shall be added to the calibration solutions.

The chromium concentration of the calibration solutions shall be adjusted to suit the sensitivity of the spectrometer used, so that the curve of absorbance as a function of concentration is a straight line.

7.4.1.2 Chromium mass fractions between 0,01 % and 2,0 %

Introduce into each of a series of seven 100 ml one-mark volumetric flasks the volumes of chromium stock solutions (4.7) or chromium standard solutions (4.8) shown in Table 1. Into each flask add 50 ml of the copper base solution (4.9). Dilute to the mark with water and mix well.

Table 1 — Calibration for chromium mass fractions between 0,010 % and 2,0 %

Chromium stock or standard solution volume		Corresponding chromium mass	Corresponding chromium concentration after final dilution	Corresponding chromium mass fraction of	
(4.8)	(4.7)			1 g	0,1 g
ml	ml	mg	mg/ml	%	%
0 ^a	—	0	0	0	0
4	—	0,10	0,001 0	0,010	
8	—	0,20	0,002 0	0,020	0,20
20	—	0,50	0,005 0	0,050	0,50
—	2	1,0	0,010	0,10	1,0
—	3	1,50	0,015	0,150	1,50
—	4	2,0	0,020	0,20	2,0

^a Blank test on reagents for calibration curve.

7.4.2 Adjustment of the atomic absorption spectrometer

Fit the chromium hollow-cathode lamp (5.3) into the atomic absorption spectrometer (5.2), switch on the current and allow to stabilize. Following the manufacturer's instructions, fit the correct burner, light the flame and allow the burner temperature to stabilize. Taking careful note of the manufacturer's instructions regarding the minimum flow rate of acetylene, aspirate the calibration solution of highest concentration of analyte and adjust the burner configuration and gas flows to obtain maximum absorbance.

7.4.3 Spectrometric measurement of the calibration solutions

Aspirate the series of calibration solutions (7.4.1.2 depending on the expected chromium content) in succession into the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration curve. Spray water through the burner after each measurement, see note.

NOTE For certain types of apparatus, instead of water it is preferable to use a solution containing the attack reagents, in the same concentrations as in the test portion solutions.

7.4.4 Establishment of the calibration curve

Establish the calibration curve using measured absorbances and corresponding analyte amounts. Use appropriate spectrometer software or off-line computer for regression calculations or prepare a graphical representation.

7.5 Determination

7.5.1 General

The analyses shall be carried out independently, in duplicate.

7.5.2 Preliminary spectrometric measurement

Carry out a preliminary measurement on the test portion solution (7.1.2 or 7.1.3) following the procedure specified in 7.4.2 and 7.4.3 at the same time as the spectrometric measurements are carried out on the calibration solutions (see 7.4.1). Estimate the preliminary analyte amount by using the calibration curve (7.4.4).